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EXPLOSIVES RESEARCH & DEVELOPMENT ESTABLISHMENT

TECHNICAL MEMORANDUM No. 7/M/50

The Physical Properties of Desensitised Nitro-Glycerine

D. V. Clifford

Waltham Abbey,
Essex.

November,
1950

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The Physical Properties of Desensitised Nitro-Glycerine

by

D.V. Clifford

This Memorandum does not contain classified information of overseas origin

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Reference: XR 510/8

1. ABSTRACT.

The specific gravity, viscosity, sensitiveness, calorimetric value, and refractive index of nitroglycerine, triacetin, and mixtures of these, with and without carbamite, have been determined. Details of methods and results are recorded.

2. INTRODUCTION.

The sensitiveness and other physical properties of nitroglycerine, desensitised with varying amounts of plasticiser, is of interest in a number of processes. The amount of published information is meagre. It was decided to carry out determinations of specific gravity, viscosity, sensitiveness, calorimetric value and refractive index on such mixtures. In order to limit the work, the only desensitiser used was triacetin. Mixtures were tested with and without 1 per cent carbamite, which for some applications is required as a stabiliser.

3. SPECIFIC GRAVITY.

3.1. Method Employed.

Accurate specific gravities of series of liquid phase mixtures have been obtained by means of a simple pyknometer constructed as shown in Fig. 1. The liquid was introduced through the side arm A by applying suction to the side arm B. After being brought to the required temperature in a thermostat bath, the distances between the menisci and the graduation marks M were measured using a cathetometer. The weight of liquid used was determined. The instrument was calibrated along the side arms by using various weights of freshly-distilled water. The nitroglycerine and triacetin used were dried by evacuation at 0.5 mm. of mercury until degassing was complete.

3.2. Results.

3.2.1. Triacetin.

A number of samples of triacetin have been analysed but only the two samples supplied by Messrs A. Boake, Roberts have been investigated, since these complied with the British Standard Specification 2D11 for Aircraft Material. The results of analysis are given in the Appendix.

/The

The specific gravities at 25°C., after vacuum-drying, of the two samples obtained from Messrs A. Boake, Roberts are given below:-

Sample	Date Received	Specific gravity 25°/25°
A	February, 1947	1.1601
B	March, 1949	1.1578

3.2.2. Nitroglycerine.

The specific gravities of nitroglycerine, determined at various temperatures, are given in Table 1, together with values already quoted in the literature (1, 2, 3).

The nitroglycerine used in the tests now reported was extracted from dynamite, filtered, and dried by prolonged evacuation at 0.5 mm. of mercury. The sample of nitroglycerine used by W.H. Perkin (1) had been prepared on a laboratory scale and dried in contact with anhydrous potassium carbonate and phosphoric anhydride. The determinations by C.G. Jackson (3) had been carried out on undried nitroglycerine.

TABLE 1

Specific Gravities of Nitroglycerine

Original	Temperature °C.						
	0/0	4/4	10/10	15/15	20/20	25/25	30/30
Current determinations (apparent)					1.596	1.591	1.588
W.H. Perkin		1.6144	1.6066	1.6009	1.5958	1.5910	
Landolt-Bornstein				1.5978			
C.G. Jackson (apparent)	1.618			1.6000			
(corrected for buoyancy)	1.6173			1.5979			

/3.2.3.

3.2.3. Nitroglycerine, Triacetin and Carbamite Mixtures.

Three series of mixtures were made up using triacetin, sample B. The first series contained no carbamite and from 0 to 100 per cent. of nitroglycerine; the second series 1 per cent. of carbamite and from 0 to 99 per cent. of nitroglycerine; and the third series 2 per cent. of carbamite and 0 to 98 per cent of nitroglycerine.

The specific gravities of these were determined at 25°C. and are given in Table 2. In Fig.2 the percentage of nitroglycerine contained in these mixtures is plotted against specific gravity.

TABLE 2

Percentage nitroglycerine.	100	80.00	60.33	39.83	20.18	0
Percentage triacetin	-	20.00	39.67	60.17	79.82	100
Specific gravity at 25°C.	1.591	1.474	1.379	1.293	1.223	1.158
Percentage nitroglycerine	99.00	79.20	59.73	39.43	19.28	0
Percentage triacetin	-	19.80	39.27	59.57	79.02	99
Percentage carbamite	1.00	1.00	1.00	1.00	1.00	1.00
Specific gravity at 25°C.	1.580	1.469	1.375	1.291	1.221	1.156
Percentage nitroglycerine	98.00	78.39	59.12	39.02	19.77	0
Percentage triacetin	-	19.61	38.88	58.98	78.23	98.00
Percentage carbamite	2.00	2.00	2.00	2.00	2.00	2.00
Specific gravity at 25°C.	1.573	1.462	1.370	1.288	1.219	1.156

The specific gravities of mixtures containing triacetin sample A were determined at more frequent intervals covering the range of N.G. content (60 - 100 per cent.) likely to be of interest. Two series of mixtures were investigated, one containing no carbamite and the other having 1 per cent. of carbamite present.

These results are given in Table 3 and are plotted as before in Fig. 3.

/TABLE 3.

TABLE 3

Percentage nitro-glycerine	100	90.33	80.04	70.16	60.10
Percentage triacetin	-	9.67	19.96	29.84	39.90
Specific gravity at 25°C.	1.591	1.532	1.475	1.426	1.381
Percentage nitro-glycerine	99	88.82	78.47	68.98	59.62
Percentage triacetin	-	10.18	20.53	30.02	39.38
Percentage carbamite	1.00	1.00	1.00	1.00	1.00
Specific gravity at 25°C.	1.580	1.521	1.466	1.420	1.376

3.3. Discussion of Results.

It can be seen from Figs. 2 and 3 that, if the specific gravity of a mixture is known, the percentage of nitroglycerine present can be estimated, within 0.2 per cent. when the proportion of carbamite present is unknown, but between 0 and 2 per cent. The curves lie closer together for mixtures containing a higher proportion of nitroglycerine so that greatest accuracy can be obtained for mixtures most likely to be required.

Simple chemical methods are established for the quantitative determination of carbamite. The control procedure for making up de-sensitised nitroglycerine on a production scale would therefore be, first to dissolve the carbamite in the triacetin, (considerable stirring is required to effect complete dissolution) and to hold this mixture in a large stock tank and check the composition by analysis. When cleared for use the desensitiser would be drawn off as required, and mixed in measured proportions with nitroglycerine. Stirring by a hand-operated wooden paddle, or by air agitation, is sufficient for this stage. After mixing, the final composition of each batch of liquid can be readily checked, on the site, by hydrometer.

4. VISCOSITY.

4.1. Method.

The viscosities measured fell within the range covered by a No.2. British Standard U-tube viscometer which was calibrated with sucrose solution.

/4.2.

4.2. Results.

4.2.1. Viscosity Temperature Coefficients of Nitroglycerine and Nitroglycerine-Triacetin Mixtures.

The viscosities of pure nitroglycerine and of a mixture of approximately equal proportions of nitroglycerine and triacetin were measured over the range of temperature likely to be practicable. The results are shown below, together with the calculated temperature coefficient of viscosity, and are represented graphically in Fig. 4.

TABLE 4

Pure Nitroglycerine

Temperature °C. = t	15	20	25	30	35
Viscosity, centistokes = ν	30.95	23.39	17.37	13.69	10.78

Temperature Range °C.	15 - 20	20 - 25	25 - 30	30 - 35
$d\nu/dt$	-1.51	-1.20	-0.73	-0.58

TABLE 5

Nitroglycerine 50.03 per cent plus Triacetin 49.97 per cent

Temperature °C. = t	15	20	25	30	35
Viscosity, centistokes = ν	41.09	29.58	20.88	16.01	12.16

Temperature Range °C.	15 - 20	20 - 25	25 - 30	30 - 35
$d\nu/dt$	-2.30	-1.74	-0.97	-0.77

/4.22.

4.2.2. Variations in Viscosity of Samples of Triacetin.

The viscosities of samples A and B supplied by Messrs A. Boake, Roberts were measured and the following figures obtained:-

Sample	Viscosity, C.S., at 25°C.
A	14.9
B	13.9

4.2.3. Viscosity of Nitroglycerine and Triacetin Mixed in Various Proportions.

Nitroglycerine was mixed with various proportions of triacetin (i) sample A and (ii) sample B and the viscosities measured at 25°C. The figures obtained are given in Table 6 below and Fig.5 shows the variation of viscosity with the proportion of nitroglycerine present.

TABLE 6

Viscosity of Nitroglycerine-Triacetin Mixtures

Percentage nitroglycerine	0	20.18	39.83	49.94	60.33	80.00	100
Percentage triacetin sample A	100	79.82	60.17	50.06	39.69	20.00	0
Viscosity centistokes at 25°C.	13.9	17.1	20.1	21.6	20.2	18.5	17.4

Percentage nitroglycerine	0	50.03	60.10	70.16	80.04	90.33	100
Percentage triacetin sample B	100	49.97	39.90	29.84	19.96	9.67	0
Viscosity centistokes at 25°C.	14.9	20.8	21.3	20.1	19.0	18.0	17.4

/4.3.

4.3. Discussion of Results.

The viscosities of nitroglycerine and also of nitroglycerine-triacetin mixtures have large temperature coefficients, so that resistance to flow decreases 2.8 to 3.4 times when the temperature is raised from 15°C. to 35°C.

The viscosities of triacetins which complies with Specification 2D11 may vary considerably from sample to sample.

The viscosities of mixtures of nitroglycerine and triacetin are higher than calculated proportionally from the viscosities of the components, due to molecular association. Maximum viscosity is obtained with approximately equal proportions of nitroglycerine and triacetin. The small proportions of carbamite required for stabilisation have little effect on the viscosities of the mixtures. Rate of flow is a function of viscosity, and the temperature coefficients of viscosity are such that rate of flow through a given channel is increased three-fold by raising the temperature from 15°C. to 35°C. For practical mixtures containing more than 50 per cent. nitroglycerine the higher the proportion of nitroglycerine the lower the viscosity. Slight contamination of the liquids investigated leads to appreciable deviations in viscosity, so that this property is unsuitable for control tests.

5. SENSITIVENESS.

5.1. Methods employed.

Impact test measurements were carried out by C.S.A.R., Woolwich and measurements using the cavity impact method were made by C.S., E.R.D.E., Waltham Abbey. All samples had been previously dried by prolonged evacuation.

5.2. Results.

The figures obtained for insensitiveness, by C.S.A.R. on the Rotter Impact Machine, are given in Table 7.

TABLE 7

Percentage Composition			Figure of Insensitiveness PA = 100	
Nitroglycerine	Triacetin	Carbamite		
100	-	-	13	
99.0	-	1.0		11
90.0	10.0	-	31	
89.2	9.8	1.0		33
80.1	19.9	-	100	
79.3	19.7	1.0		100
60.3	39.7	-	120	
59.7	39.3	1.0		120

By the same method the following figures are obtained as standards:-

Picric acid	100
D.E.G.N.	126
C.E.	56

Results obtained by C.S., E.R.D.E., using the Bowden Fall-Hammer Cavity-Impact Machine are given in Table 8.

TABLE 8

Percentage Composition			Maximum energy at which no explosions occurred	Minimum energy at which 100% explosions occurred
Nitroglycerine	Triacetin	Carbamite		
100	-	-	125 ergs.	500 ergs.
99.0	-	1.0	125 "	500 "
90.3	9.7	-	175 "	2,250 "
88.8	10.2	1.0	1,000 "	3,000 "
80.0	20.0	-	> 4,000 "	?
78.5	20.5	1.0	> 4,000 "	?

5.3. Discussion of Results.

Mixtures containing up to 80 per cent. nitroglycerine may be handled with about the same degree of safety as D.E.G.N. or picric acid and may therefore be used on a production scale with no more risk than arises in the handling of D.E.G.N.

6. CALORIMETRIC VALUE.

6.1. Methods.

All measurements were carried out by C.S.A.R., Woolwich.

The calorimetric values of mixtures of nitroglycerine, triacetin and carbamite have not been determined directly because these mixtures cannot be ignited satisfactorily in the calorimetric bomb. Also, a residue is left in the bomb when cordite is burnt in contact with liquid triacetin.

It was found that satisfactory determinations could be made with gels produced by mixing nitrocellulose (12.2% N) with the desensitised mixture under examination, and the following procedure was therefore adopted.

/Nitrocellulose

Nitrocellulose (12.2% N) was weighed into a specimen tube 2 inches long and 3/4 inch diameter and dried to constant weight. The liquid was added in vacuo, so that a fairly homogeneous gel was obtained, and the weight of added liquid was determined. The tube containing the gel was loaded directly into the bomb; the water equivalent of the glass container was allowed for in the calculation.

6.2. Results.

TABLE 9

Percentage Composition				Cals/ gram W.L.	Intrinsic cooling Figure for triacetin Cals./1% added
Nitroglycerine	Nitrocellulose 12.2% N	Triacetin	Carbamite		
75.17	24.83	-	-	1554	
73.62	25.64	-	0.74	1507	
66.33	25.31	7.61	0.75	1295	10.0
58.02	26.06	15.18	0.74	1080	10.1
52.22	24.30	22.72	0.76	869	10.8
44.62	25.19	29.46	0.73	737	8.6

6.3. Discussion of Results.

The intrinsic cooling figure for triacetin obtained by this method does not agree with the figure 12.84 quoted in U.S. reports. The result of the last determination (737 cals./gm.) would be vitiated by methane formation etc. and should be ignored. An average figure of 10.3 calories cooling for one per cent. of triacetin may be taken.

7. REFRACTIVE INDEX.

It was decided that, since the chemical compositions of the ingredients used in the mixtures may vary slightly refractive index measurement would have no useful application.

8. ACKNOWLEDGMENTS.

This work was initiated and supervised by Mr. W.N. Hewson.

/9.

9. BIBLIOGRAPHY.

1. W.H. Perkin, Trans. Chem. Soc., 55, 685.

2. Landolt-Bornstein, Eq. Bd. IIa, p. 246.

3. C.G. Jackson, E.R.D.E., Woolwich, unpublished communication.

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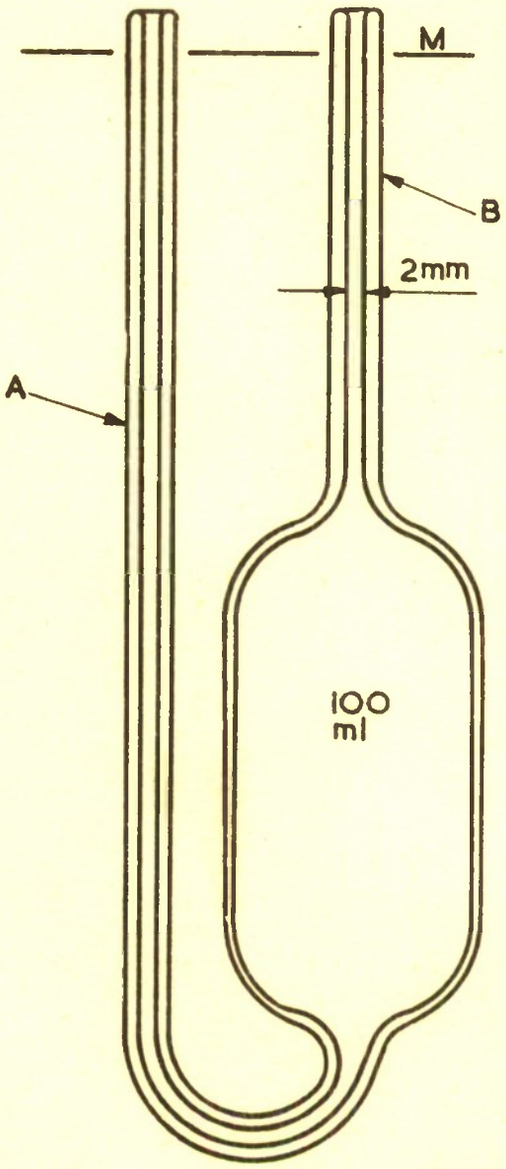
M.No.498/50

APPENDIX

Analysis of Triacetin Samples

Details of Sample	Specification Requirements	Sample 1. from A.Boake, Roberts To Specn. 2D11. Received Feb. '49.	Sample 2. from B.D.H. (Woolwich Stores) Date June '46.	Sample 3. from B.D.H. Half gal. sample Received Feb. '49.	Sample 4. from A.Boake, Roberts Received Feb. '47.	Sample 5. from Judex Chemicals (Gen. Chem. and Pharmaceutical Co. Received Mar. '49.
Description	Clear colourless liquid.	Clear colourless slightly scented.	Clear, very pale yellow. Slight odour.	Clear, colourless. Slight odour.	Clear, slight yellow tinge. Slight odour.	Clear, colourless. Slight odour.
Specific Gravity at 15°C.	1.160 - 1.170	1.168	1.175	1.168	1.169	1.170
Acidity (1) % $CH_3.COOH$ by wt.	1.15%	0.04%	0.28%	0.02%	1.14%	0.08%
(2) To methyl orange.	Not acid	Neutral	Alkaline*	Neutral	Acid	Acid
Ash.	0.02%	0.003%	0.11%	0.002%	0.001%	0.002%
Freedom from glycerol and water.	Free	Free	Free	Free	Free	Free
Acetyl content as $CH_3.COOH$.	80.0%	80.15%	76.42%	81.15%	78.42%	79.15%

* Repeat using B.D.H. Universal Indicator gave pH of approximately 5.0.



FULL SIZE.

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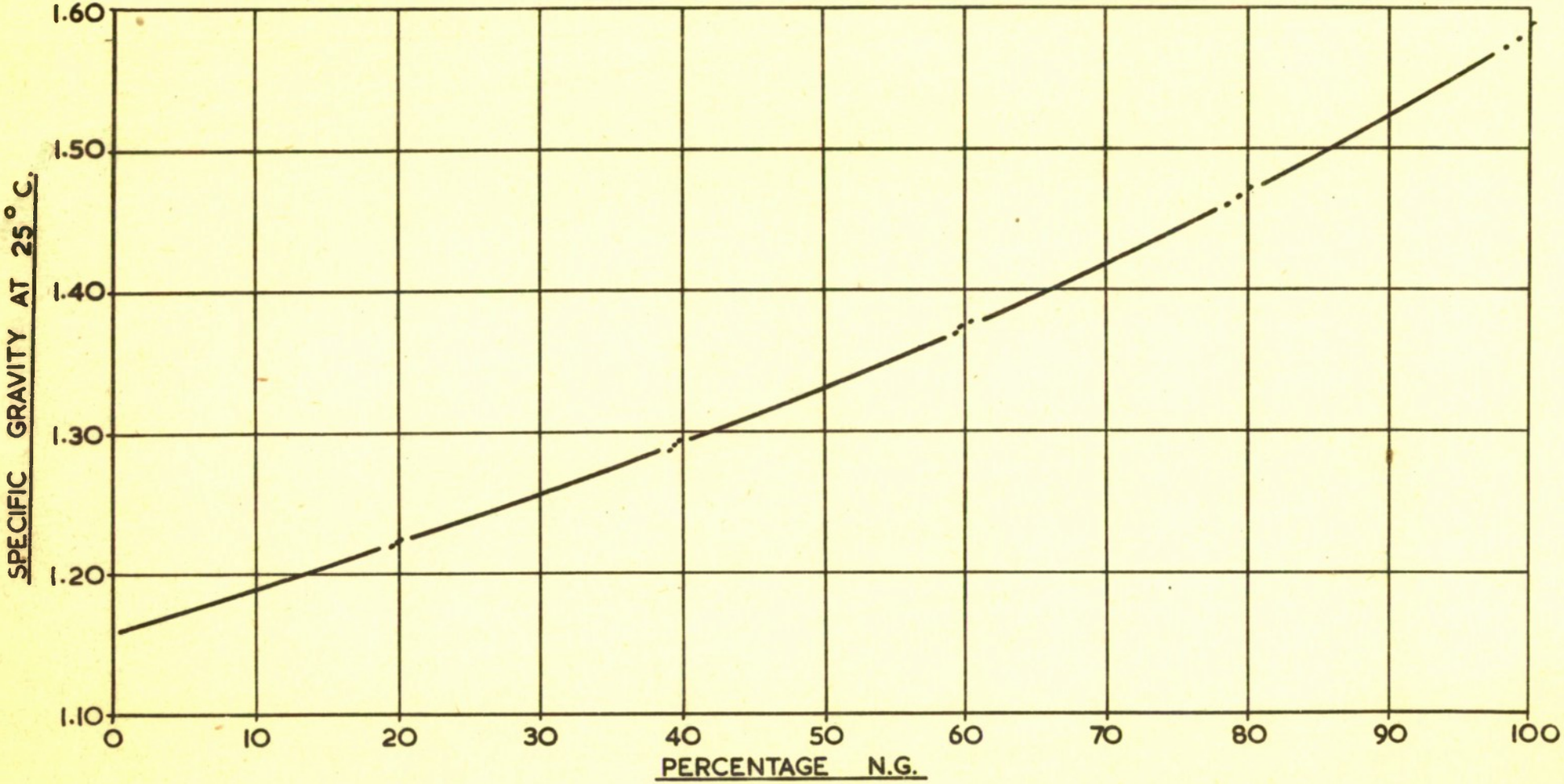
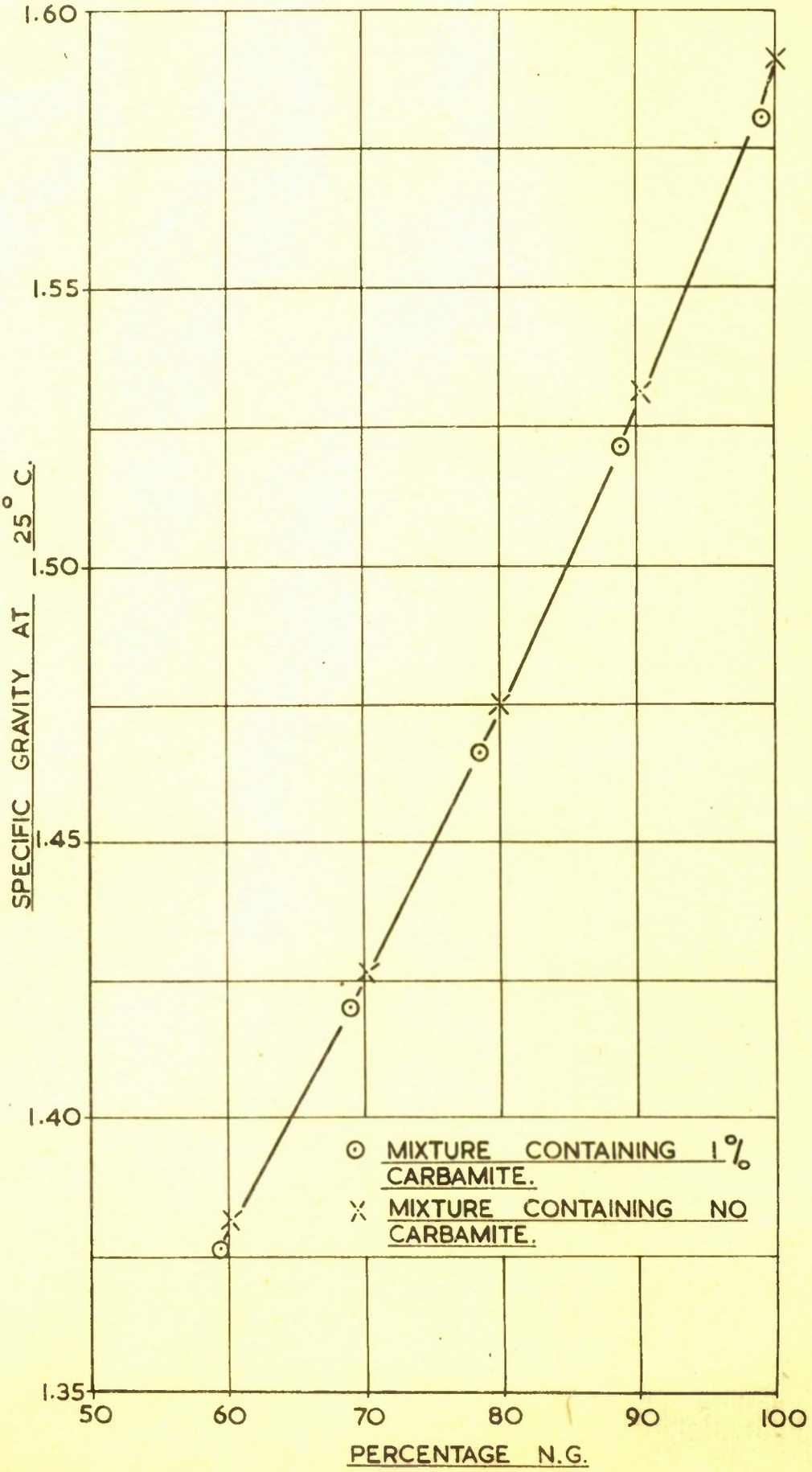
FIG. 1.



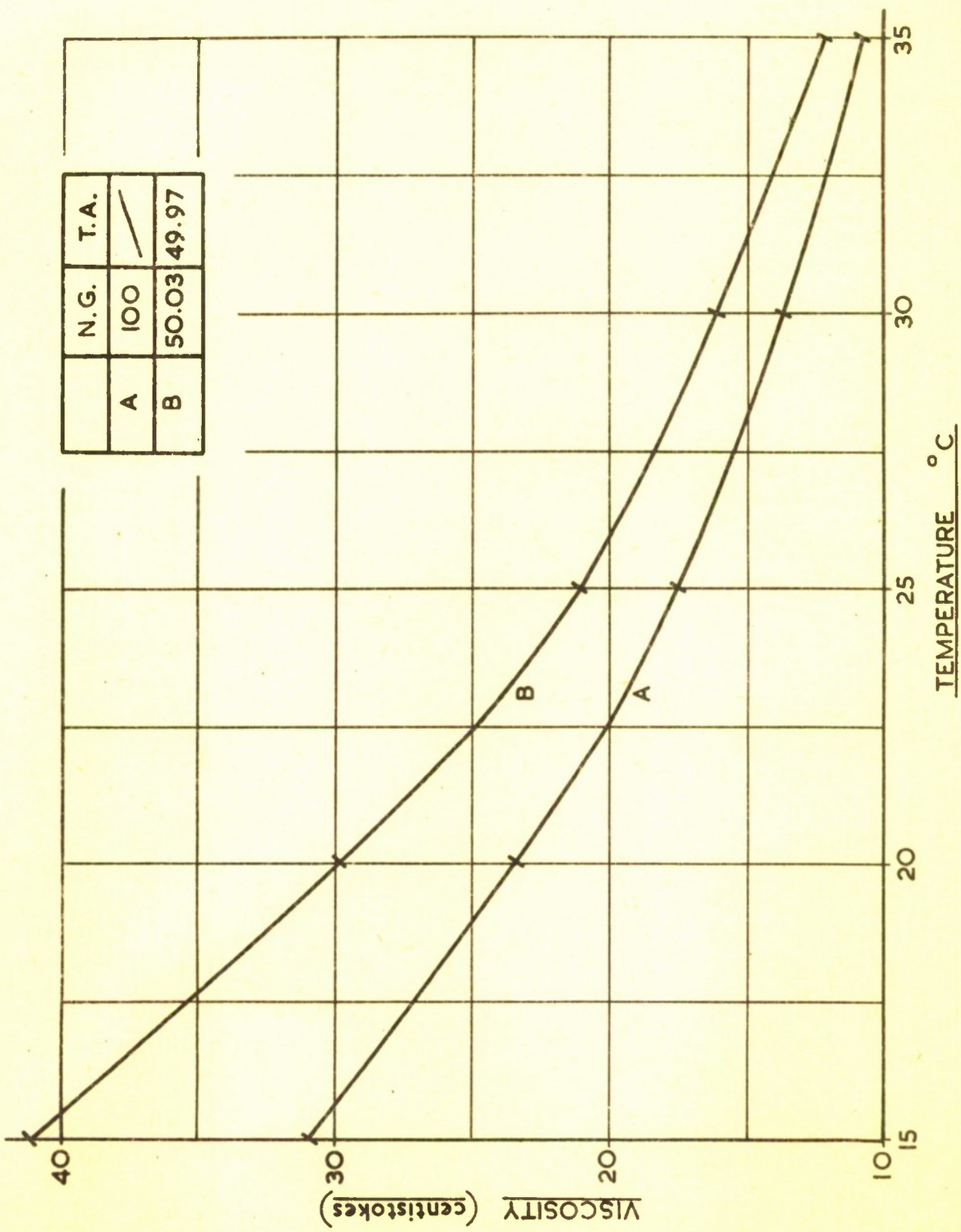
SPECIFIC GRAVITIES OF MIXTURES OF NITROGLYCERINE TRIACETIN AND CARBAMITE.

FIG. 2.



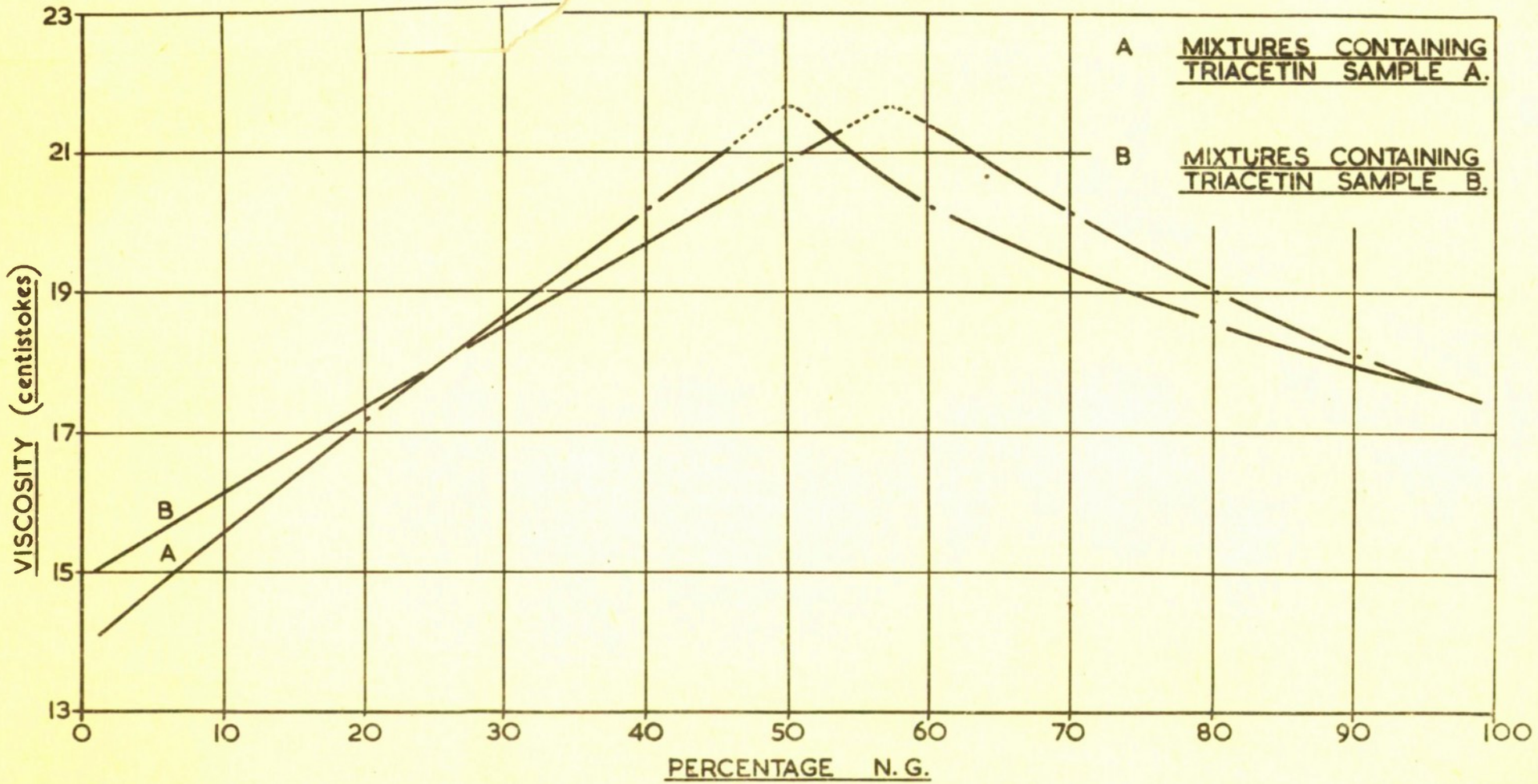
SPECIFIC GRAVITIES OF MIXTURES OF NITROGLYCERINE CARBAMITE AND TRIACETIN CONTAINING GREATER THAN 50 % NITROGLYCERINE.

FIG. 3.



VARIATION OF VISCOSITY COEFFICIENT WITH TEMPERATURE.

FIG. 4.



VISCOSITY COEFFICIENTS OF NITROGLYCERINE TRIACETIN MIXTURES AT 25° C.

FIG. 5.

[dstl]

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